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271-507

RDTR NO. 92

March 1967

BUNDER STUDY - VISUAL CAST FLAME

H. R. WAITE and Y. ARIKAWA
Ordnance Research Incorporated

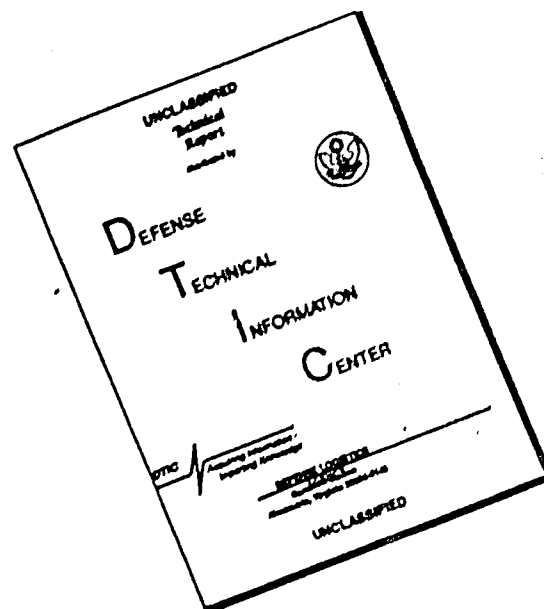
This work was sponsored jointly by the U. S. Air Force and the U. S. Navy. Funds were provided by the Air Force Armament Laboratory, Illumination Branch, Eglin Air Force Base, Florida, by Eglin AIFR PG-4-58, and the Naval Air Systems Command, Armament Division, Pyrotechnic Section, Washington, D. C. This report will also be included in the final report on AIFR-PG-4-58.

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U. S. Naval Ammunition Depot
Crane, Indiana 47522

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The report was reviewed for adequacy and technical accuracy by B. E. Douda.

Released

BH Calkins

B. H. Calkins, Manager
Concept Development Division
Research and Development Department

Final Summary Report

BINDER STUDY - VISUAL CAST FLARES

N00164-67-C-0086

Submitted To

**U. S. Naval Ammunition Depot
Research and Development Department
Crane, Indiana**

By

**Ordnance Research Incorporated
11 Miracle Strip Parkway, S. W.
Fort Walton Beach, Florida**

27 March 1967

ABSTRACT AND SUMMARY

Techniques and castable formulations were developed that produce luminous efficiencies between 42,000 and 45,000 candle-seconds/gram; no diameter effect was apparent in the range of 1.5 to 4.95 inches. The early experimental effort on the use of compounded polyesters and urethanes was discontinued in favor of the use of glycidyl methacrylate and similar vinyl monomers. This class of monomer gives the desired solvation of the perchlorate (with concomitant increase in viscosity of the binder for improved particle wetting) and a controlled exotherm in large diameter castings. Final physical properties are attained after 24-hour post-cure. The use of sodium perchlorate in lieu of the insoluble sodium nitrate yielded a casting slurry of lower viscosity at constant binder percentage; the use of sodium nitrate was therefore discontinued. The use of a trimodal distribution of magnesium improved casting ease at the 15 percent binder level.

Early program effort concentrated on the use of kraft paper cases in order to simulate standard flare hardware. The recognized "tunnel effect" reduced the luminance values obtained and masked differences attributable to the binder type and content. Composition efficiencies were then obtained on free standing grains, displacement cast and the cylindrical surfaces inhibited. The program has demonstrated the feasibility of cast illuminating flares based on commercial materials.

INTRODUCTION

Previous experimentation on cast illuminating flares has centered on two techniques: the incorporation of an increased percentage of binder in the magnesium-sodium nitrate system to impart enough fluidity for casting followed by a cure or polymerization to a solid grain, or the use of mixed oxidizer salts, such as sodium and calcium nitrates, to form a eutectic mixture of melting point low enough for hot pouring followed by cooling to a solid column. The efficiency of the former system is significantly lower due to the additional binder; the safety and ease of fabrication of the latter system is not completely satisfactory. The pyrotechnic laboratory at Picatinny Arsenal established a number of years ago that there is a simple linear relationship between the decrease in luminous efficiency and the percentage Laminac-Lupersol binder in the pressed system. The percentage of binder is now generally held to the minimum necessary for adequate compressive strength for this reason. This loss is the direct result of increased radiation in the infrared, organic binders being oxidized to water vapor and carbon dioxide which are strong emitters at 1.8, 2.7 and 4.3 microns. The decrease in flame temperature due to this increased radiant heat loss shifts the radiation spectrum to shorter wavelengths and decreases markedly emittance in the visible.

The amount of binder necessary to impart sufficient fluidity for casting is on the order of 12 to 15 percent with a greater concomitant increase in the loss of luminous efficiency. An improvement in the solids loading (solids loading being the ratio of total solids, both fuel and oxidizer, to the total mass of liquid) will improve luminous efficiency. Our experimental effort has been concentrated on the technique of the use of soluble inorganic perchlorate oxidizers in place of the solid nitrate oxidizers employed in the pressed system.

The use of soluble oxidizers allows a lower percentage of binder to yield a unit of luminous efficiency comparable to the magnesium-sodium nitrate pressed system. The composition 49 percent atomized magnesium, 36 percent sodium perchlorate dissolved in 12 percent diethylene glycol, and 3 percent carbon black gellant is a thixotropic fluid. Combustion of this liquid composition in a simple end-burning configuration in a plastic case gave a luminous efficiency of 40,000 candle-seconds/gram. The liquid visual flare system may be polymerized to a solid cast grain by reaction of the polyfunctional alcohol with diisocyanates to yield in situ a polyurethane binder. Polyesters represent another class of monomer candidate; the reported solubility of inorganic perchlorate in low molecular weight esters indicates that acrylic and methacrylic ester monomers are logical candidates.

POLYESTER BINDER SYSTEMS

An examination of the literature on available unsaturated polyester systems, such as the commonly employed "Laminac 4116", shows that they are solutions of high molecular weight condensation products of dibasic acids and bifunctional alcohols in a solvent/monomer which is usually styrene for reasons of economy. Twelve to 15 percent styrene is required for cross-linking all polyester double bonds but amounts up to 50 percent are added to increase the flexibility and impact resistance of the cured resin. This percentage of added hydrocarbon, however, decreases markedly the solubility of inorganic salts in the resin. Only silver perchlorate has appreciable solubility in hydrocarbon solvents with greatest solubilities in aromatic solvents such as toluene and benzene (undoubtedly also styrene which is vinylbenzene). Hazards associated with the handling of anhydrous silver perchlorate and the raw material cost preclude its use as a soluble oxidizer. The solubility of other inorganic perchlorates is dependent on the oxygen content of the organic solvent and the types of functional groups represented. The usual order of solvent power is hydroxyl > carbonyl > ether.

Solubilities of Inorganic Perchlorates, Grams/100 Grams Solvent, 25°C¹

Cation	Ethylene Glycol	Ethyl Alcohol	Acetone	Ethyl Acetate	Ethyl Ether
Sodium	95.5	14.71	51.75	9.65	Insol.
Magnesium		23.96	42.89	70.91	0.29
Strontium		180.66	150.06	136.93	Insol.
Barium		124.62	124.67	112.95	Insol.
Lithium		151.76	136.52	95.12	113.72

The raw polyester syrup or solid polymer not admixed with styrene is the product of a condensation reaction between acids and alcohols; the cross-linking agent may be chosen from the following oxygen-containing monomers: allyl alcohol, methyl acrylate, methyl methacrylate, vinyl acetate, ethyl acrylate, and ethyl methacrylate. Specialty polyester resins based on components having large percentages of bound oxygen and good solvent action for inorganic perchlorates may be prepared by laboratory combinations of these monomers; all laboratory efforts were based on commercially available starting materials so that the results of this effort will be directly useable in large-scale production. The binder studies were limited to those with maximum oxygen content.

A literature survey was conducted to determine the types of polyester resins available. Manufacturers of basic polyester resins were contacted; however, the majority of replies received indicated that the commercially available polyester formulations contained either styrene or vinyl toluene and were therefore unsuitable for use as a flare binder material. One basic polyester resin, Aropol 7200, was acquired for initial investigation. Several commercially available basic monomers were selected to study their compatibility with Aropol 7200. Among these found compatible were methyl methacrylate (MeMa), methyl acrylate (MaA), and 2-hydroxyethyl methacrylate (2-HEMA), which offered an hydroxyl group to aid in dissolving partially or entirely oxidizers such as sodium perchlorate, increasing the solids to binder loading ratio. A formulation containing approximately 20 percent binder (2 percent Aropol 7200 and 18 percent MeMa) and equal parts of oxidizer and fuel was blended. Visual inspection of the mixture revealed that the binder failed to polymerize completely and that the resultant mixture was a crumbly agglomerate of fuel and oxidizer. Apparently most the methyl methacrylate monomer, having a low vapor pressure, vaporized during the polymerization exotherm. Since difficulty was encountered in utilizing only MeMa as a binder material, grains were fabricated using 2-HEMA with a small amount of MeMa to reduce viscosity and increase wetting of solid particles. The technique employed was to add the oxidizer (NaClO₄) incrementally to the liquid monomer to dissolve as much of the oxidizer in the monomer as possible. As the oxidizer dissolved in the monomer, the solution increased in viscosity until finally (after all the oxidizer was added) a

heavy paste formed. MeMa was added to reduce viscosity to facilitate the addition of the solid magnesium fuel. Grains to be tested were cast in paper tube flare cases approximately 1.5 inches I.D. and 2.5 inches long. To prevent possible flash-by and to obtain adhesion of the binder material to the walls of the flare case, all cases were lined with a polyester base liner material, similar to the Laminac liner now currently used in certain pressed flare systems. Intensity measurements were made with Edgerton, Germeshausen & Grier Radiometer System, Model 580, with spectral response in the 350 and 550 millimicron wavelength range. The test results of these grains (as reported under CVF-3) show a low radiant intensity of 20,200 candle-seconds/gram. The binder content of this grain was 25 percent; a further reduction in binder content will increase the radiant intensity.

ACRYLIC ESTER BINDER SYSTEMS

Further analysis of 2-hydroxyethyl methacrylate as a binder material was undertaken based on the solubility of sodium perchlorate in this monomer and the observed oxidizer particle wetting. The use of polyester resins was temporarily discontinued in order to concentrate efforts on the development of cast systems incorporating minimum binder. In attempts to cast grains of lower binder content, difficulty was encountered due to a spontaneous binder material polymerization reaction. Experiments were conducted to verify the cause of this behavior. A sample prepared by adding atomized magnesium to a binder sample, 2-HEMA, reacted to the gel stage almost immediately and produced a hard porous polymer within 30 minutes. Atomized aluminum powder gave no reaction under identical conditions. It was then concluded that the magnesium was responsible for spontaneous monomer polymerization and not perchlorate; a behavior in contrast to the action of strontium perchlorate in activating the polymerization of methyl methacrylate. The exact mechanism of the reaction was not known but it was believed that the methacrylic acid, present in the monomer about 3 percent, reacted with the magnesium to initiate the polymerization reaction. Magnesium and other monomers not containing methacrylic acid failed to show signs of reaction. By working rapidly, without adding catalyst to the binder monomer, grains were cast using 13 to 17 percent binder. The technique employed was to add the perchlorate to the binder, followed immediately by the magnesium with rapid mixing and immediate casting into the flare cases. The flare composition cured in about ten minutes. These flares were test fired and relative luminous efficiencies were recorded. The luminous efficiency for the 17 percent binder grain was 33,600 candle-seconds/gram and the 13 percent binder grain was 41,600 candle-seconds/gram (CVF-10 and CVF-11).

In attempts to cast flares using dimethylaminoethyl methacrylate, difficulty was encountered in initiating the polymerization reaction by peroxide catalysts, although it has been reported that polymerization has been achieved through the use of these catalysts. The recommended

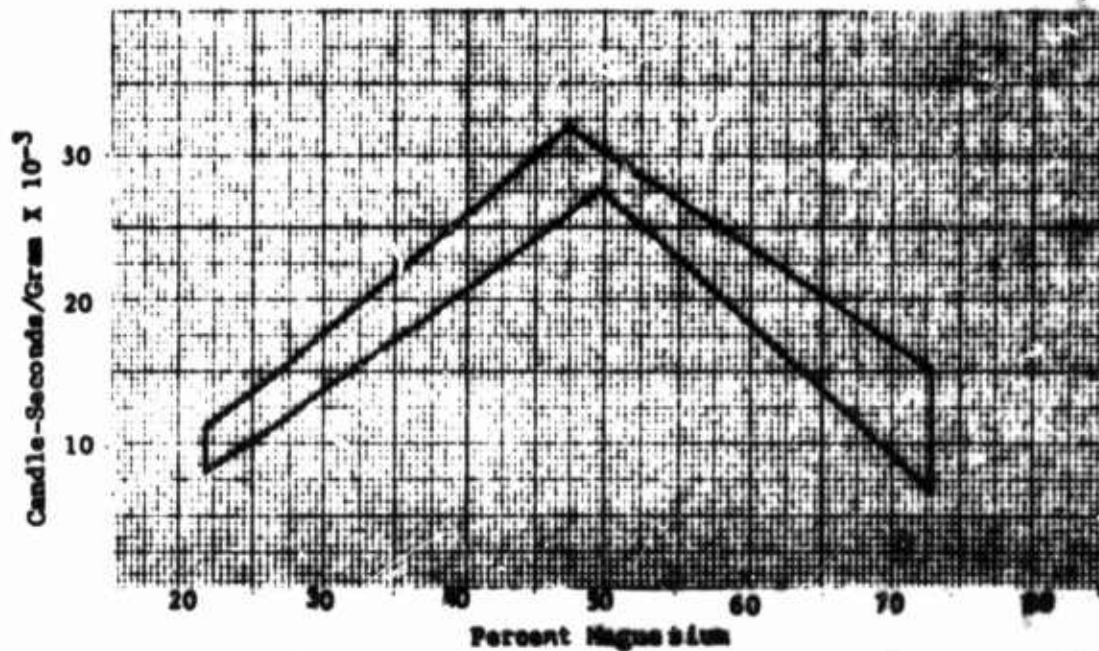
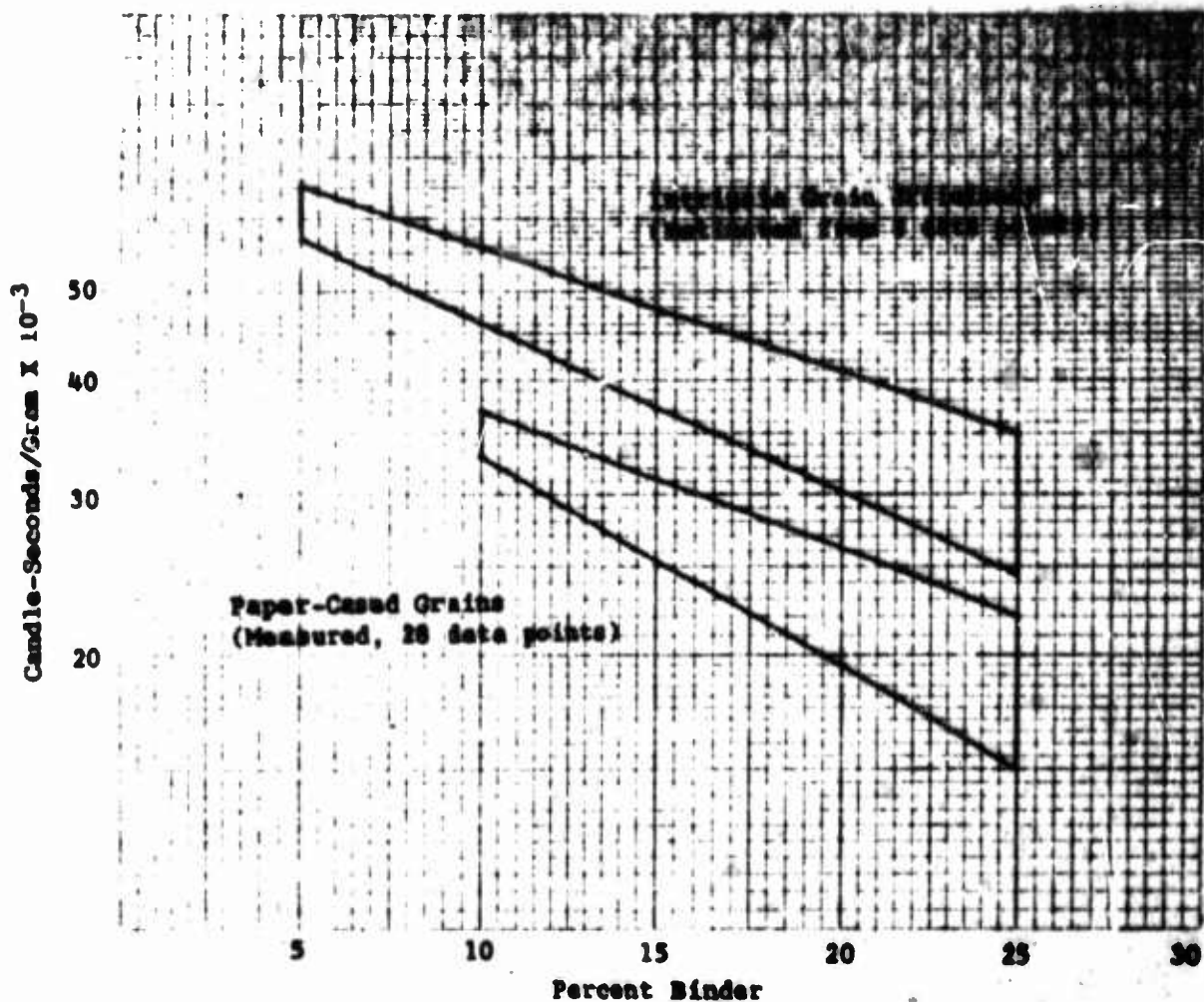
catalysts are also compounds which are not affected by the monomer tertiary amine group; none were available at the time the flares were fabricated. Attempts to cast grains utilizing dimethylaminoethyl methacrylate were suspended until suitable polymerization catalysts were obtained; the solvation of perchlorates by amino groups makes this class of binder attractive.

Glycidyl acrylate (37.5 percent oxygen) and glycidyl methacrylate (33.7 percent oxygen) appear to dissolve more sodium perchlorate than 2-hydroxyethyl methacrylate (37.0 percent oxygen). Greater fluidity was observed for compositions prepared with glycidyl methacrylate facilitating casting. No reaction with atomized magnesium was observed with this binder system and the polymerization is easily controlled with the usual catalysts and promoters. In accordance with discussions with the Program Manager, Mr. Bernard Douba, effort was concentrated on the development of a binder system suitable for controlled production batch mixing and casting at NAD crane for larger scale testing.

The effect of binder percentage and type in cast flare compositions was investigated in order to correlate binder percentage and luminous efficiency. Lower binder percentages yield higher luminous efficiencies for all compositions; the accompanying graph shows the relationship for grains cast in paper cases. On the basis of later experimental effort on aluminum-cased and free-standing grains in the range of binder percentage between 15 and 20 percent, the estimated intrinsic luminous efficiency is indicated. In the case of 2-hydroxyethyl methacrylate (2-HEMA), 15 percent was the lowest binder concentration that could be incorporated in the magnesium- NaClO_4 composition and still maintain grain integrity. Glycidyl methacrylate permitted a minimum of 10 percent binder while maintaining grain integrity.

Compositions containing LiClO_4 and NaClO_4 in equal proportions exhibited lower luminous efficiencies than one component oxidizer 2-HEMA binder compositions. The decreased free sodium concentration in the flare plume offset the increased composition oxygen percentage. In an attempt to incorporate $\text{Mg}(\text{ClO}_4)_2$ into cast compositions, the mixture ignited when $\text{Mg}(\text{ClO}_4)_2$ was added to glycidyl methacrylate (no magnesium was present). The reaction between $\text{Mg}(\text{ClO}_4)_2$ and glycidyl methacrylate or glycidyl acrylate is apparently hypergolic, the acrylate being more reactive than the methacrylate. The following graph summarizes the effect of the oxidizer/fuel ratio for paper-cased compositions. Compositions outside a narrow range exhibited sharply lower values of luminous efficiency. Using 15 percent 2-HEMA in the magnesium/ NaClO_4 reaction system, flares were fabricated and tested. The results are plotted as an almost symmetrical curve about the 42.5 percent axis (oxidizer/fuel ratio of 1:1). It is apparent that the luminous efficiencies of Mg/NaClO_4 compositions are more sensitive to reaction stoichiometry than pressed magnesium/ NaNO_3 compositions.

Effect Of Binder Percentage Concentration
On Luminous Efficiency Of Cast Visual Plates



Effect Of Magnesium On Luminous Efficiency Of Fifteen
Percent Binder Compositions (Paper Cased)

Glycidyl acrylate compositions produced luminous efficiencies comparable to glycidyl methacrylate compositions for identical binder percentages. Ethylene dimethacrylate (32.3 percent oxygen) compositions yielded a luminous efficiency comparable to that produced by other 15 percent binder systems. Highly-oxygenated binders produce equivalent values of luminous efficiency; processing characteristics determine the utility of the binder system. Polymerization of these monomer systems was effected by dissolving 1 percent benzoyl peroxide in the monomer. A room-temperature curing agent, N,N Dimethyl p-toluidine or Promoter 402 (American Cyanamid), was used in the amount of 60 mg per 100 grams of composition to accelerate polymerization. To insure complete polymerization, the grains were subjected to a post cure temperature of 75°C for 24 hours, minimum, before testing.

To compare the relative effects of sodium nitrate and sodium perchlorate as oxidizers on the luminous efficiency of visual flare compositions, two grains were cast under identical conditions, i.e., 15.0 percent binder and an O/F ratio of 1:1. Since sodium perchlorate partially dissolved in the binder, glycidyl methacrylate, the resulting composition was "wetter" in the unpolymerized state than the composition prepared using sodium nitrate. Sodium nitrate, being relatively insoluble in typical binder monomers, posed problems of producing a slurry sufficiently wet to facilitate casting. In addition, the sodium nitrate formulation, not producing a viscous monomer due to the insolubility of the nitrate in the monomer, presented additional problems of the monomer separating from the solids. Hence, in a grain cast upright, the lower portion would have a tendency to be monomer-rich. A somewhat similar problem was posed by sodium perchlorate with low-viscosity binders but not as extreme as in the case of sodium nitrate. The luminous efficiencies of sodium perchlorate and sodium nitrate flare grains were identical within the limits of experimental error.

In order to measure the luminous efficiencies as closely as possible to the intrinsic value for each composition, an attempt was made to cast grains free from the case effect (or "chimney effect") which seemed inherent in earlier units which were cast in paper cases. One method is to produce free-standing grains by displacement casting. Whether the units cast in paper cases performed as well as those cast as free-standing grains was investigated. One set of flares, CVP-79A to CVP-79D, was cast as free-standing grains, inhibited with two thin coats of a polyester along the surfaces ordinarily inhibited by the paper case. Another identical set was cast in paper cases, CVP-80A to CVP-80D. As shown in the Appendix, the free-standing grains clearly produced higher luminous efficiencies. In addition, intensity-time traces of the free-standing grains revealed a more uniform and consistent performance in contrast to those grains cast in paper cases.

In order to establish a final formulation for recommendation to the sponsoring agency, 1500-gram flare grains were cast and tested to determine the effect of increased burning surface area. Compositions previously tested were evaluated on the basis of a 1.5 inch burn surface diameter. Experimental flare CVP-89, containing 16 percent binder, was displacement cast and inhibited with a laminating polyester resin along the surfaces ordinarily inhibited by the case. The diameter of the grain was approximately 4.75 inches and the grain height was approximately 3.5 inches; the burn time of the grain was 41.0 seconds (burn rate of .085 in/sec) and the luminous efficiency measured was 41,200 candle-seconds/gram. The luminous efficiency of this larger grain is equal to the efficiencies produced by the smaller, 1.5 inch diameter grains. As indicated in the appendix, a trimodal distribution of magnesium powder was utilized in the fabrication of experimental flare CVP-89. Since sodium perchlorate is slightly soluble in the binder material, no attempt was made to utilize a mixed particle size distribution of oxidizer. The smaller diameter grains tested early in the program contained either a single particle size of magnesium (CVP-79 contained 22 μ magnesium only) or a bimodal distribution of magnesium (CVP-73 contained both 22 μ and 125 μ magnesium). A mixed particle fuel composition did not seem to affect the luminous efficiency; therefore, a trimodal distribution of magnesium (22 μ , 125 μ , and 200 μ) was incorporated to facilitate blending and casting. Another unit, CVP-90 was cast incorporating magnesium powder conforming to Specification JAN 382, Type III. The luminous efficiency calculated from the intensity-time trace was 25,800, the low value due to partial smoke obscuration; a value of 39,000 candle-seconds/gram was estimated for this grain.

MISCELLANEOUS BINDER SYSTEMS

A 1500-gram grain incorporating a fluoroheptyl methacrylate binder was cast with some difficulty due to the insolubility of sodium perchlorate in the binder. No luminous efficiency calculations were made for this grain due to complete smoke obscuration. It appeared that this particular composition produced excessive smoke in comparison to the compositions based on the oxygenated acrylic esters. An attempt was made to cast flares using a commercial urethane binder, Coast Pro-Seal 796-80. Difficulty was encountered in dispersing the perchlorate and the magnesium uniformly in the binder due to its high viscosity (greater than 10,000 cps). Binder percentages lower than 18 percent were not feasible. The in situ reaction of tolylene di-isocyanate with diethylene glycol or low molecular weight polyethylene glycols also gave viscous binder solutions. Fifteen percent General Electric Silicone RTV-8111 as a binder gave a composition that resembled the glycidyl methacrylate-sodium nitrate formulations in that the solids were not sufficiently wetted by the binder to facilitate casting. The luminous efficiency of this composition fell into the same range as the other 15 percent binder compositions. From the standpoint of casting ease the glycidyl methacrylate/sodium perchlorate composition is superior and the Silicone RTV/sodium perchlorate and glycidyl methacrylate/sodium nitrate systems are about equal.

CONCLUSIONS AND RECOMMENDATIONS

Castable illuminating compositions have been developed and demonstrated with efficiencies between 40,000 and 45,000 candle-seconds/gram based on the use of a number of highly oxygenated monomers. It is recommended that more endothermic binder/monomer systems be investigated, i.e., those containing either nitro- or difluoroamino- functional groups. The limitation on the solids loading that can be achieved with any binder system and blending and fill technique suggests that the use of an oxidizer/binder is necessary for increased efficiency in cast systems.

Throughout the entire research program, no attempt was made to cast grains using vacuum techniques. The brevity of time plus the emphasis on binder systems precluded the study of utilizing vacuum techniques either before or during casting of flare compositions. It is believed that the application of vacuum to the composition either before or during casting will result in better physical properties and higher luminance values. A study of the advantages or disadvantages of utilizing vacuum casting seems desirable. In an attempt to derive intrinsic luminous efficiency values of the cast compositions investigated, some effort was expended in avoiding cast effects. The methodology employed in developing cast flare grains creates possible areas of study associated with flare case design hitherto restricted by the pressed grain systems.

APPENDIX

CVF-3

Luminous Efficiency

2-Hydroxyethyl Methacrylate	17.00 percent	20,200 candle-seconds/gram
Arapol 7200	3.40	
Methyl Methacrylate	4.75	
NaClO ₄	40.80	
Chaff (A1)	.30	
Magnesium	34.00	

CVF-73

Glycidyl Methacrylate	10.0 percent	A - 25,000 candle-seconds/gram
Sodium Perchlorate	45.0	B - 21,400
Magnesium 22 μ	22.5	C - 28,800
Magnesium 125 μ	22.5	D - 31,300
		E - 28,000

CVF-79

Glycidyl Methacrylate	15.0 percent	A - 37,600* Candle-seconds/gram
Sodium Perchlorate	42.5	B - 41,600
Magnesium 22 μ	42.5	C - 34,600
		D - 22,600*

CVF-80

Glycidyl Methacrylate	15.0 percent	A - 31,800 candle-seconds/gram
Sodium Perchlorate	42.5	B - 29,350
Magnesium 22 μ	42.5	C - 29,790
		D - 26,040

CVF-89

Glycidyl Methacrylate	16.0	41,200 candle-seconds/gram
Sodium Perchlorate (over 200 mesh)	42.0	
Magnesium 22 μ (MIL-P-14067A)	14.0	
Magnesium 125 μ (MIL-P-14067A)	14.0	
Magnesium 200 μ (MIL-P-14067A)	14.0	

* Partial Smoke Obscuration

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Security Classification

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1. ORIGINATING ACTIVITY (Corporate method) Ordnance Research Incorporated 11 Miracle Strip Parkway, S.W. Fort Walton Beach, Florida		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	
		2b. GROUP	
3. REPORT TITLE BINDER STUDY - VISUAL CAST FLARES			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (Last name, first name, initial) Waite, Dr. Hal R. and Y. Arikawa			
6. REPORT DATE March 1967		7a. TOTAL NO. OF PAGES 15	7b. NO. OF REFS 69
8a. CONTRACT OR GRANT NO. Eglin MIPR-PG-6-58		8b. ORIGINATOR'S REPORT NUMBER(S) Final Summary Report of 27 March 1967	
a. PROJECT NO. N00164-67-C-0086		8c. OTHER REPORT NO(S) (Any other numbers that may be used/used this report)	
9. AVAILABILITY/INTIMATION NOTICES Distribution of this document is unlimited.			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Air Force Armament Laboratory, Eglin AFB USMAC Crane, Indiana	
13. ABSTRACT Techniques and castable formulations were developed that produce luminous efficiencies between 42,000 and 45,000 candle-seconds/gram; no diameter effect was apparent in the range of 1.5 to 4.95 inches. The early experimental effort on the use of compounded polyesters and urethanes was discontinued in favor of the use of glycidyl methacrylate and similar vinyl monomers. This class of monomer gives the desired solvation of the perchlorate (with concomitant increase in viscosity of the binder for improved particle wetting) and a controlled exotherm in large diameter castings. Final physical properties are attained after 24-hour post-cure. The use of sodium perchlorate in lieu of the insoluble sodium nitrate yielded a casting slurry of lower viscosity at constant binder percentage; the use of sodium nitrate was therefore discontinued. The use of a trimodal distribution of magnesium improved casting ease at the 15 percent binder level. Early program effort concentrated on the use of kraft paper cases in order to simulate standard flare hardware. The recognized "tunnel effect" reduced the luminance values obtained and masked differences attributable to the binder type and content. Composition efficiencies were then obtained on free standing grains, displacement cast and the cylindrical surfaces inhibited. The program has demonstrated the feasibility of cast illuminating flares based on commercial materials.			

DD FORM 1473

1 JAN 64

0101-007-0000

UNCLASSIFIED

Security Classification

KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Binder study Acrylates Illuminating compositions Monomers Perchlorates Solubilities Graft flame Methacrylates						

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